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# Electrolyte and Electrode Passivation for Thin Film Batteries

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# Overview

- Motivation for research
- Experimental
- Results
  - Impedance spectroscopy
  - DC breakdown
  - XPS
  - XRD
- Alternative passivation films
- Qualitative trends
- Summary



# Motivation

- Provide chemical stability at anode for high conductivity, low reductive stability electrolytes.
- Provide chemical stability at cathode for high conductivity, low oxidative stability electrolytes.
- Identify robust passivation film tolerant to humid air or wet processing for multi-step patterning of thin film batteries.
- Examine if other Li electrolytes could be nitrided.



# Experimental

## Film Preparation:

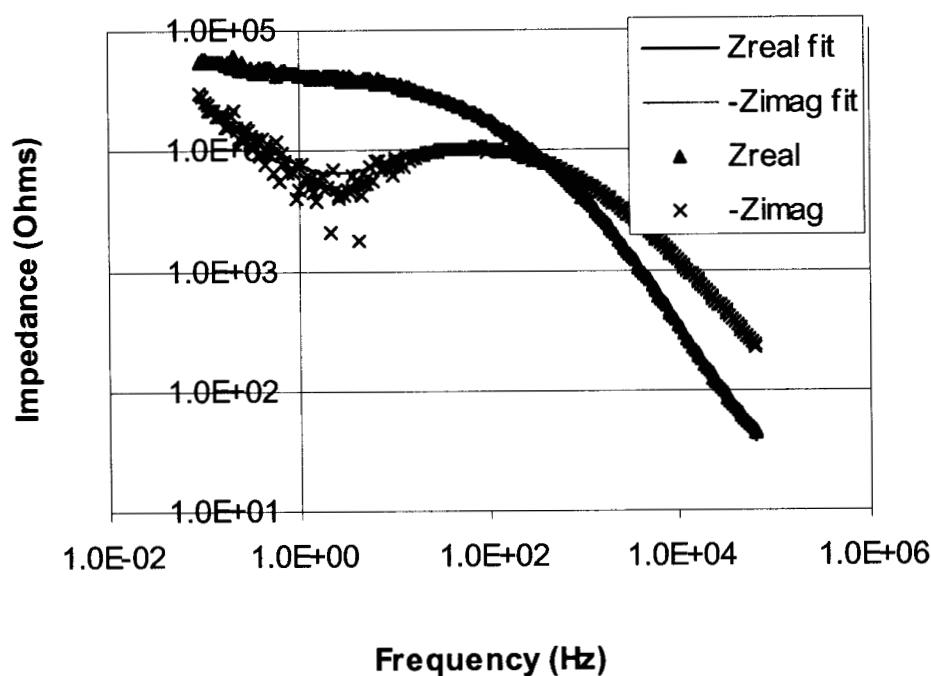
- RF sputtered from  $\text{Li}_2\text{CO}_3$  target
- Power levels of 75-200 W for 3" target
- Sputter gas: blends of  $\text{CO}_2$ ,  $\text{O}_2$ ,  $\text{N}_2$ , Ar
- Li electrodes thermally evaporated

## Characterization:

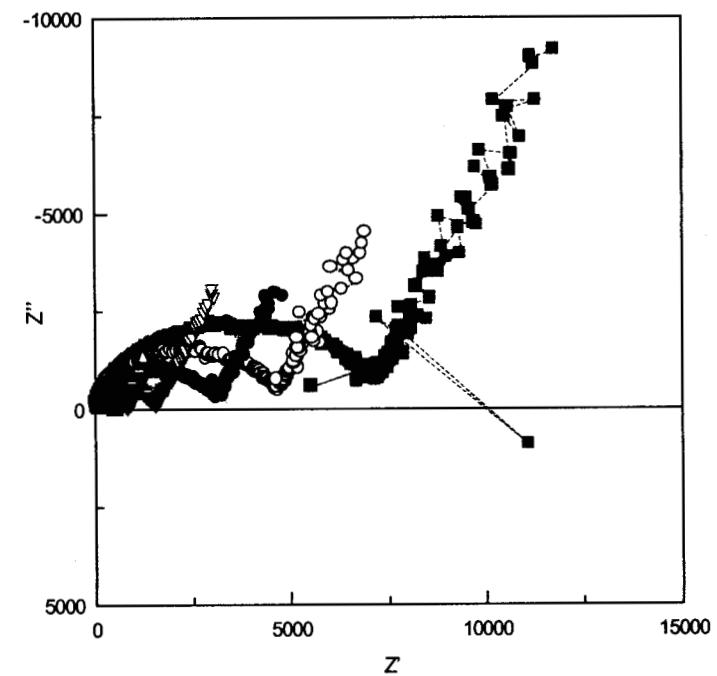
- Impedance spectroscopy
- DC breakdown
- XPS
- XRD
- TEM



# Impedance Spectroscopy Results



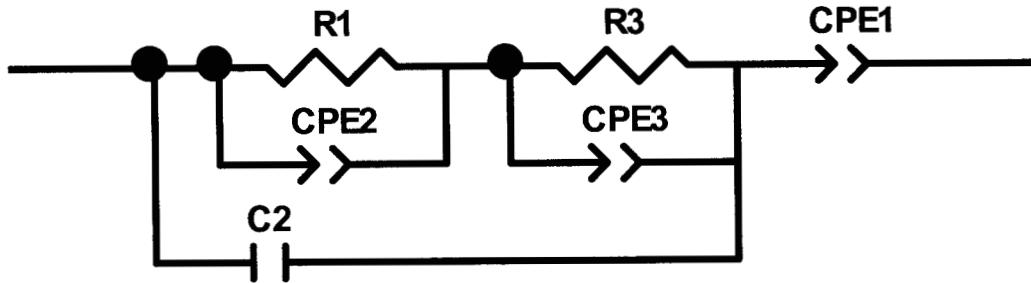
Bode plot of  $\text{Mo}|\text{Li}_2\text{CO}_3|\text{Mo}$



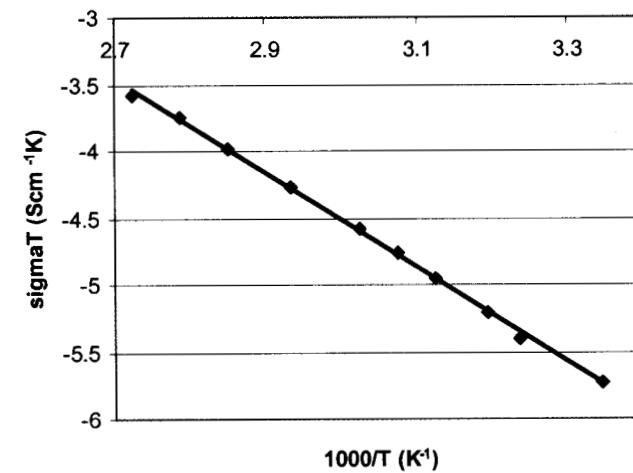
Complex plane plots of  $\text{Mo}|\text{Li}_2\text{CO}_3|\text{Mo}$



# Impedance Spectroscopy Results



Equivalent Circuit Model

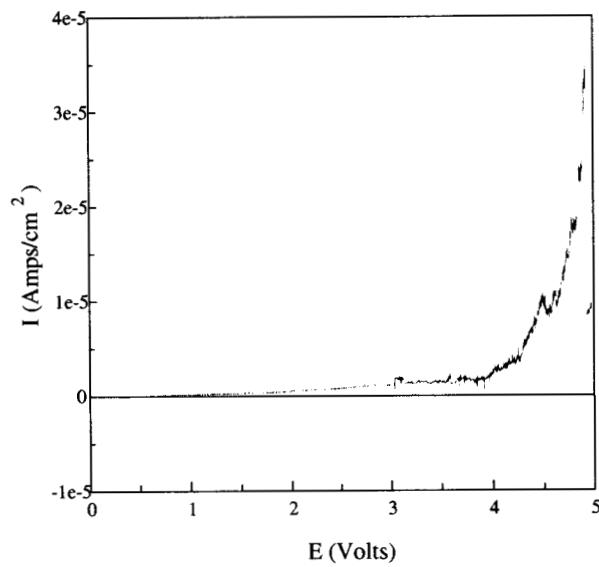


Arrhenius plot of Mo|Li<sub>2</sub>CO<sub>3</sub>|Mo

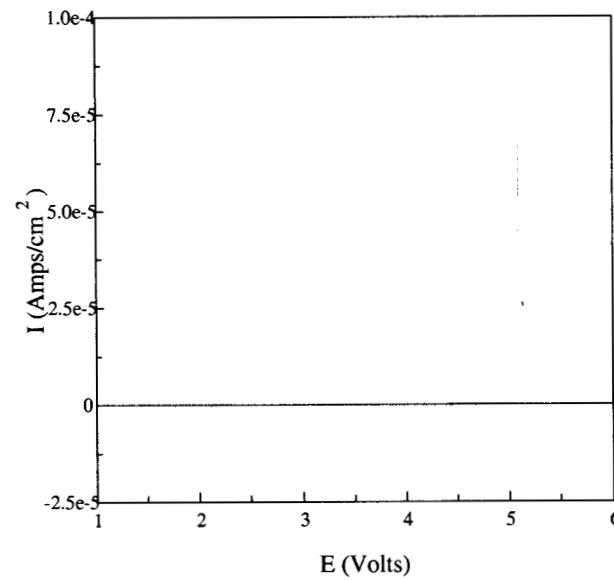


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# DC Characterization Results



DC breakdown of Mo|Li<sub>2</sub>CO<sub>3</sub>|Mo

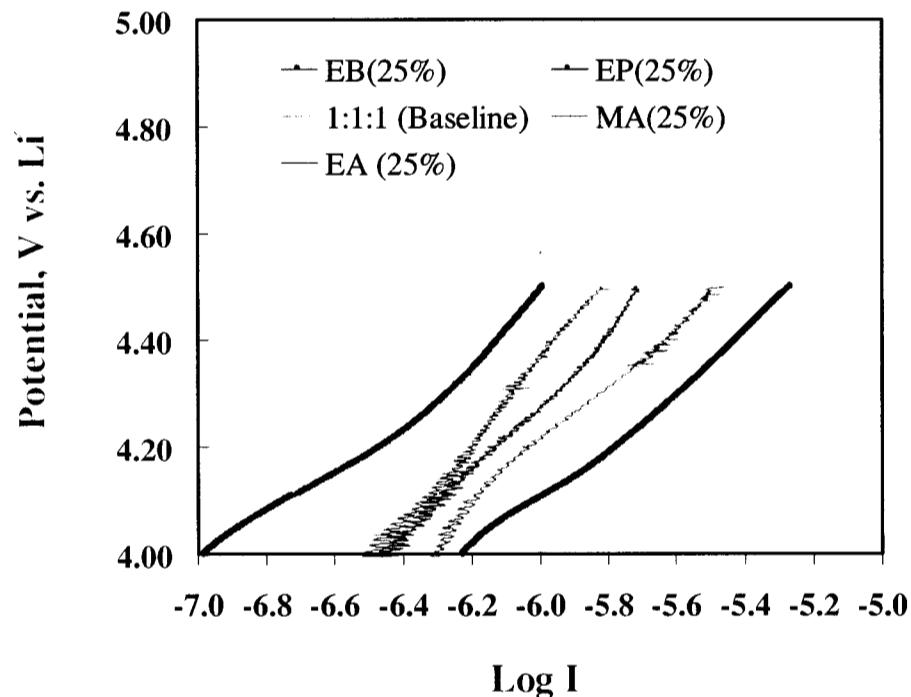


DC breakdown of Mo|Lipon|Mo



# DC Characterization Results

Tafel Plots corresponding to Electrolyte Oxidation



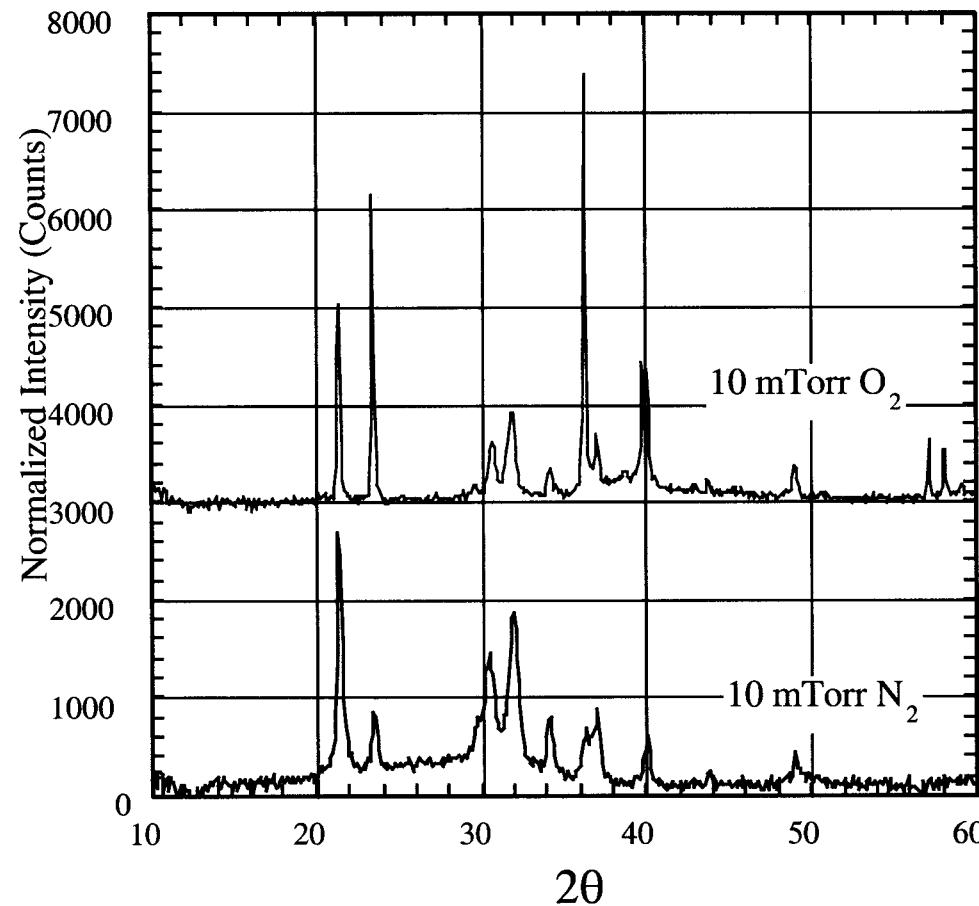
Tafel plots of various liquid electrolytes



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# X-ray Diffraction: $\text{Li}_2\text{CO}_3$ Sputtered in $\text{N}_2$ and $\text{O}_2$

Collected at Stanford Synchrotron Radiation Laboratory



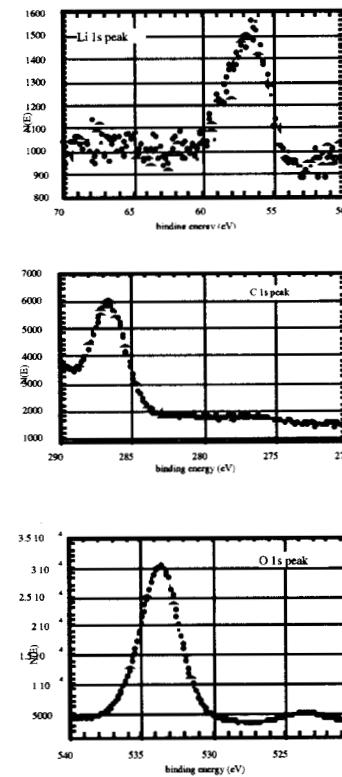
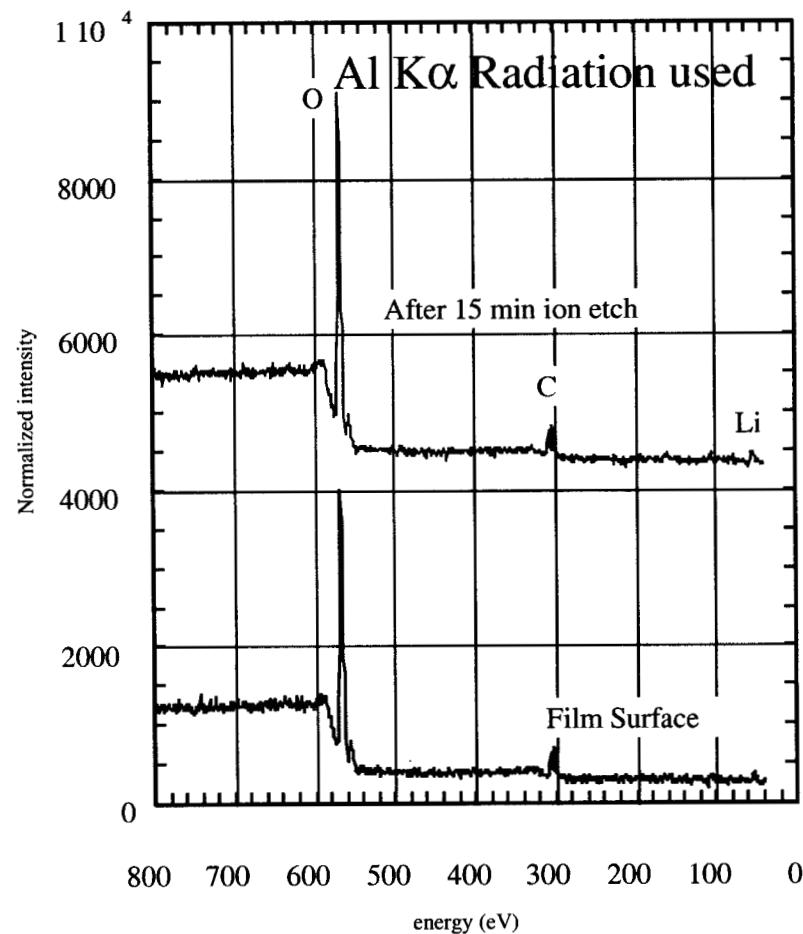
- Crystalline  $\text{Li}_2\text{CO}_3$  Observed, Grain size  $\sim 20$  nm



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# X-ray Photoelectron Spectroscopy

$\text{Li}_2\text{CO}_3$  Sputtered in 100%  $\text{O}_2$



$\text{Li}_4$

C

$\text{O}_{3.7}$

- ~1/1 Combination of  $\text{Li}_2\text{CO}_3$  &  $\text{Li}_2\text{O}$

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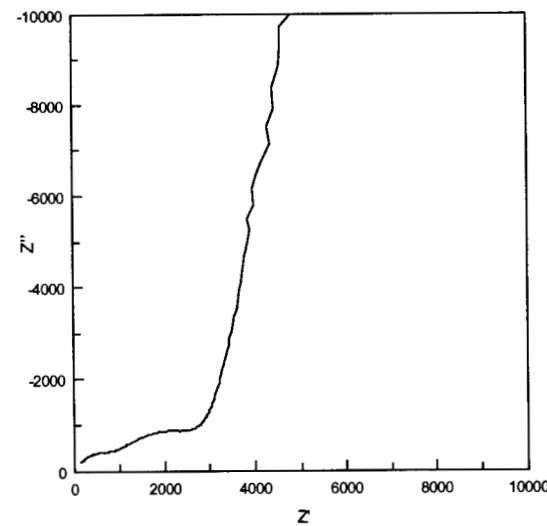
## Qualitative Trends

- Increase in air stability, repeatability with increasing power (less Li<sub>2</sub>O)
- Increase in target decomposition, bonding failure with increasing power
- Little variation on film properties with sputter gas composition
- Conditions yielding low deposition rate favored resputtering

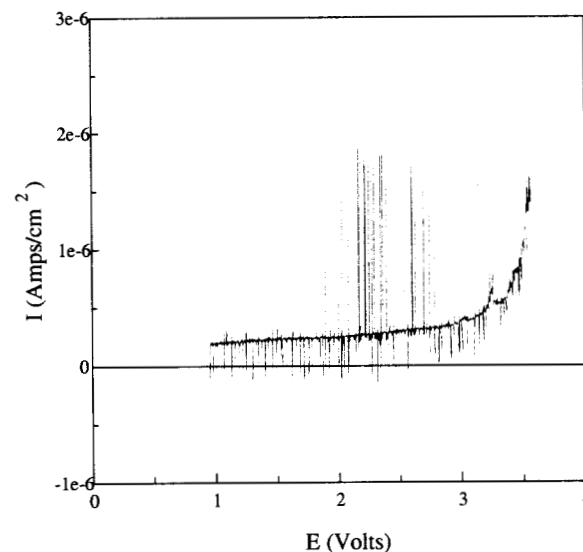


# Alternative Passivation Films

- LiF: poor conductivity
- LiF/Li<sub>3</sub>PO<sub>4</sub> (Ar/O<sub>2</sub> sputter gas) poor oxidative stability
- LiF/Li<sub>2</sub>CO<sub>3</sub> poor air stability/target stability



LiF/Li<sub>3</sub>PO<sub>4</sub> film



LiF/Li<sub>3</sub>PO<sub>4</sub> film



## Summary

- Passivation films for improved anodic and cathodic protection were examined.
- Films are to be used in conjunction with high conductivity electrolytes.
- $\text{Li}_2\text{CO}_3$  films prepared via sputtering bear  $\text{Li}_2\text{O}$ .
- Fully amorphous films could not be obtained.
- RF sputter power level chiefly determined film stability, while sputter gas composition did not change film properties.
- Increased RF power resulted in higher stability films, but also resulted in target degradation